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SPECIFICATION FOR NICKEL CHLORIDE HEXAHYDRATE, PESTICIDAL GRADE

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Indian Standard

SPECIFICATION FOR NICKEL CHLORIDE HEXAHYDRATE, PESTICIDAL GRADE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 October 1980, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Nickel chloride hexahydrate is used as a general fungicide on agriculture crops.

0.3 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for nickel chloride hexahydrate, pesticidal grade.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of greenish crystals, free from extraneous matter.

2.2 The material shall also comply with the requirements given in Table 1.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR NICKEL CHLORIDE HEXAHYDRATE

(*Clauses* 2.2 *and* 5.1)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO APPENDIX
(1)	(2)	(3)	(4)
i)	Nickel (as Ni) content, percent by mass, *Min*	23·7	A
ii)	Cobalt (as Co) content, percent by mass, *Max*	0·5	B
iii)	*p*H of aqueous solution, *Min*	3·75	C
iv)	Acid insoluble matter, percent by mass, *Max*	0·05	D
v)	Iron (as Fe) content, percent by mass, *Max*	0·02	E

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirements given in IS : 8190 (Part I)-1980*.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to the provisions of the Insecticides Act and Rules:

a) Name of the material;
b) Name of manufacturer;
c) Date of manufacture;
d) Batch No;
e) Net mass of contents;
f) Nickel content, percent (*m*/*m*); and
g) Minimum cautionary notice as worded in the Insecticides Act and Rules.

3.2.1 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

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*Requirements for packing of pesticides: Part I Solid pesticides (*first revision*).

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in the 'Indian standard Methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Till such time the standard under preparation is published, samples shall be drawn as agreed to between the concerned parties.

5. TESTS

5.1 Tests shall be carried out as prescribed in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF NICKEL (as Ni) CONTENT

A-1. PRINCIPLE

A-1.1 Nickel is determined by gravimetric method using dimethylglyoxime as precipitating agent.

A-2. REAGENTS

A-2.1 Dilute Sulphuric Acid — 10 percent (*v/v*).

A-2.2 Tartaric Acid

A-2.3 Ammonium Hydroxide — Relative density 0·90.

A-2.4 Dimethylglyoxime Solution — 1 percent (*m/v*) solution in alchohol.

A-3. PROCEDURE

A-3.1 Weigh accurately about 2 g of the crushed and well-mixed material. Dissolve in water, add 2 ml of dilute sulphuric acid and dilute to 250 ml. Take a 25-ml aliquot, add 0·5 g of tartaric acid, neutralize with ammonium hydroxide, and then make just acidic with dilute sulphuric acid adding 10 drops in excess. Dilute to 250 ml. Heat to 70°C and add 30 ml of dimethylglyoxime solution. Add ammonium

---

*Specification for water for general laboratory use (*second revision*).

hydroxide dropwise, until smell of ammonia persists, then add 5 ml of ammonium hydroxide and stir well. Allow the precipitate to settle and the solution to cool to 25 to 30°C. Filter through a sintered glass crucible (G No. 3). Wash the precipitate with water (at about 25°C) till free from chlorides. Dry the crucible at 110 to 120°C to a constant mass.

A-4. CALCULATION

A-4.1 Nickel (as Ni) content, percent by mass $= \frac{203{\cdot}2\, M_1}{M_2}$

where

M_1 = mass in g of the precipitate, and
M_2 = mass in g of the material taken for the test.

APPENDIX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF COBALT (as Co) CONTENT

B-1. PRINCIPLE — Cobalt is determined by iodometric method.

B-2. REAGENTS

B-2.1 Potassium Hydroxide Solution — 10 percent (*m/v*).

B-2.2 Acetic Acid —Glacial.

B-2.3 Potassium Nitrite Solution — 50 percent (*m/v*).

B-2.4 Ammonium Nitrate Solution — 5 percent (*m/v*).

B-2.5 Standard Potassium Permanganate Solution — 0·1 N freshly standardized.

B-2.6 Dilute Sulphuric Acid — 10 percent (*v/v*).

B-2.7 Potassium Iodide

B-2.8 Standard Sodium Thiosulphate Solution — 0·1 N.

B-2.9 Starch Indicator Solution

B-3. PROCEDURE

B-3.1 Weigh accurately about 1 g of the material, dissolve in 30 ml of water and minimum volume of dilute sulphuric acid, add 1 ml of potassium hydroxide solution, then add 2 ml of glacial acetic acid and heat to boiling. Add 10 ml of potassium nitrite solution, stir and digest on a steam-bath for 30 minutes. Allow to stand overnight. Filter through a sintered glass crucible (G No. 4) and wash with small quantities of cold ammonium nitrate solution containing 1 percent by volume of glacial acetic acid until free from nitrite. Place the crucible in the beaker in which precipitation was carried out, add exactly 50 ml of standard potassium permanganate solution and 25 ml of dilute sulphuric acid, and then add sufficient water to cover the crucible. Heat to about 50°C until the yellow precipitate has dissolved, then cool and add 1 g of potassium iodide. Titrate the liberated iodine against standard sodium thiosulphate solution, using starch as indicator.

B-3.2 Carry out a blank titration with the same volumes of standard potassium permanganate solution and dilute sulphuric acid and potassium iodide in the same manner as with the sample.

B-4. CALCULATION

B-4.1 Cobalt (as Co) content, percent by mass $= \frac{0{\cdot}536\, N\, (V_1 - V)}{M}$

where

N = normality of standard sodium thiosulphate solution;

V_1 = volume in ml of standard sodium thiosulphate solution used in the blank titration;

V = volume in ml of standard sodium thiosulphate solution used in the titration with the material; and

M = mass in g of the material taken for the test.

APPENDIX C

[*Table* 1, *Item* (iii)]

DETERMINATION OF *p*H OF AQUEOUS SOLUTION

C-1. APPARATUS

C-1.1 *p*H Meter — with glass electrode.

C-2. PROCEDURE

C-2.1 Place 5 g of the material in freshly boiled and cooled distilled water, dilute to 25 ml and mix. Determine the *p*H value of the solution with the *p*H meter at 25°C.

APPENDIX D

[*Table* 1, *Item* (iv)]

DETERMINATION OF ACID INSOLUBLE MATTER

D-1. PRINCIPLE

D-1.1 The material is dissolved in acidified water and the residue left on filtering is weighed.

D-2. REAGENTS

D-2.1 Dilute Hydrochloric Acid — 10 percent (*v/v*).

D-3. PROCEDURE

D-3.1 Weigh accurately about 10 g of the material and dissolve in 125 ml of water and 1 ml of dilute hydrochloric acid. Heat the solution to boiling. Filter through a weighed and prepared Gooch crucible, or sintered glass crucible (G No. 4) and wash the residue thoroughly with hot water. Dry the crucible at 110 ± 5°C to a constant mass.

D-4. CALCULATION

D-4.1 Acid insoluble matter, percent by mass $= \dfrac{100\,M_1}{M}$

where

M_1 = mass in g of the residue, and

M = mass in g of the material taken for the test.

APPENDIX E

[*Table* 1, *Item* (v)]

DETERMINATION OF IRON (as Fe) CONTENT

E-1. PRINCIPLE

E-1.1 Iron (as Fe) is estimated by colorimetric method, using ammonium thiocyanate.

E-2. APPARATUS

E-2.1 Nessler's Cylinders — 50-ml capacity.

E-3. REAGENTS

E-3.1 Concentrated Hydrochloric Acid

E-3.2 Concentrated Nitric Acid

E-3.3 Ammonium Hydroxide — 1 : 4 (*v/v*).

E-3.4 Dilute Ammonium Hydroxide — 1 : 10 (*v/v*).

E-3.5 Dilute Hydrochloric Acid — approximately 4 N.

E-3.6 Ammonium Persulphate

E-3.7 Ammonium Thiocyanate Solution — approximately 5 percent (*m/v*).

E-3.8 Standard Iron Solution — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2SO_4.6H_2O$] in about 100 ml water containing 10 ml of dilute sulphuric acid 10 percent (*v/v*) and dilute to 1 000 ml. One ml of the solution will contain 0·1 mg of iron (as Fe).

E-4. PROCEDURE

E-4.1 Dissolve exactly 1 g of the material in 20 ml of water. Add 2 ml of concentrated hydrochloric acid and 5 drops of concentrated nitric acid. Heat to boiling and add sufficient ammonium hydroxide to precipitate completely. Filter and wash the precipitate with dilute ammonium hydroxide until the washings are colourless. Dissolve any precipitate on the filter paper in 5 ml of hot dilute hydrochloric acid and dilute with water to about 20 ml. Transfer to a Nessler cylinder and dilute to the mark. Add about 30 mg of ammonium persulphate and 3 ml of ammonium thiocyanate solution and mix.

E-4.2 Carry out a control test in the other Nessler cylinder, proceeding as with the material, using 2 ml of standard iron solution. The limits prescribed in the requirements shall be taken as having not been exceeded, if the intensity of colour produced with the material is not greater than that produced in the control test.

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